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13. ABSTRACT (Maximum 200 words)

Silicon nitride is a potential candidate for several high temperature structural applications, like heat engine components, cutting tool inserts, bearings, and wear parts. In use, the ceramic component is often subject to dynamic loading conditions at elevated temperatures. Hence, it is necessary to evaluate the performance of the material under dynamic loading conditions over a range of temperatures.

This research project focused on the behavior of *in situ* reinforced silicon nitride, under dynamic compressive loading, over a range of temperatures. Novel experimental (the Split Hopkinson Compression Bar) and analytical (Differential Strain Measurement) techniques have been developed to aid the evaluation of the dynamic properties of *in situ* reinforced silicon nitride over a range of temperatures. The properties (primarily, the elastic modulus and the failure stress) of the material has been studied from room temperature to 1000°C. The experimental work has been supplemented by microstructural studies, both before and after testing, in order to gain an insight into the mechanisms of failure.

14. SUBJECT TERMS
In-situ reinforced silicon nitride, high strain-rate, microstructure, boundary phases

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Enclosure 1

FINAL TECHNICAL PROGRESS REPORT

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1. STATEMENT OF PROBLEM STUDIED

Silicon nitride is a potential candidate for several high temperature structural applications, like heat engine components, cutting tool inserts, bearings, and wear parts. In use, the ceramic component is often subject to dynamic loading conditions at elevated temperatures. Hence, it is necessary to evaluate the performance of the material under dynamic loading conditions over a range of temperatures.

This research project focused on the behavior of *in situ* reinforced silicon nitride, under dynamic compressive loading, over a range of temperatures. Novel experimental (the Split Hopkinson Compression Bar) and analytical (Differential Strain Measurement) techniques have been developed to aid the evaluation of the dynamic properties of *in situ* reinforced silicon nitride over a range of temperatures. The properties (primarily, the elastic modulus and the failure stress) of the material has been studied from room temperature to 1000°C. The experimental work has been supplemented by microstructural studies, both before and after testing, in order to gain an insight into the mechanisms of failure.

2. SUMMARY OF THE MOST IMPORTANT RESULTS

2.1 The Microstructure and Boundary Phases of *In-Situ* Reinforced Silicon Nitride (published manuscript)

M. Liu and S. Nemat-Nasser

Abstract: The microstructure of an *in-situ* reinforced silicon nitride, gas pressure sintered with La_2O_3, Y_2O_3, and SrO additives and then heat treated, is examined with X-ray diffraction, SEM, and high resolution TEM. Two crystalline rare-earth apatite phases, $La_5Si_3O_{12}N$ and $Y_5Si_3O_{12}N$, are identified at the grain pockets and at the two-grain boundaries. The thickness of the crystalline phases at the two-grain boundaries is approximately 1.7nm, in compliance with the suggested equilibrium intergranular spacing. A glassy phase is also present at the grain pockets and at the two-grain boundaries due to incomplete crystallization of the boundary phases. The thickness of the amorphous phase at the two-grain boundaries varies from 0.7nm to 3.0nm, suggesting that compositional inhomogeneities exist at these areas. Based on the microstructural observations, the structures of the crystalline boundary phases, the equilibrium intergranular film thickness, and the mechanisms resulting in incomplete recrystallization of the glassy phase in the *in-situ* reinforced silicon nitride are discussed in this research paper.

2.2 Microstructure of a Bearing-Grade Silicon Nitride (submitted manuscript)

M. Liu and S. Nemat-Nasser.

Abstract: The microstructure of a bearing-grade silicon nitride, pressurelessly sintered with TiO_2, Y_2O_3, and AlN additives and then hot-isostatically pressed, is examined with high-resolution

TEM, SEM, and XRD. The material consists of large acicular β-Si_3N_4 grains and small equiaxed α-Si_3N_4 grains. An amorphous phase containing the sintering aids is observed to exist at the two-grain boundaries and at the grain pockets. No crystalline phase is identified. The α-to-β and β-to-β grain boundaries appear straight and well defined. (1010) and (1120) are the dominant crystalline planes observed at the β-grain boundaries. The intergranular spacing of the two-grain boundaries is 1.0nm for the high contrast boundary phase and is 0.8nm when a low-contrast boundary phase is present, confirming that the film thickness is strongly dependent on the boundary-phase composition. The α-to-α boundaries are often curved and the thickness of the amorphous film at these boundaries varies from 0.7 to 1.1nm. Evidence of near-intimate contact between β-grains is also observed.

2.3 Dynamic Properties of *in situ* Reinforced Silicon Nitride at Elevated Temperatures (manuscript in-press)

G. Raghavendra, S. Nemat-Nasser and M. Liu

Abstract: This study is aimed at evaluating the compressive dynamic response of *in situ* reinforced silicon nitride at elevated temperatures. The material has been characterized before experimentation microstructurally. A unique experimental facility, the modified split Hopkinson pressure bar, has been developed for the purpose of conducting high strain-rate tests at elevated temperatures. A new measurement technique, called Differential Strain Measurement (DSM) has been developed to obtain accurate values of strain. Extensive tests show that the elastic modulus and the failure stress of the material decreases with increasing temperature.

(Refer to **Appendix A** for full paper)

2.4 High Temperature Testing of In-Situ Toughened Silicon Nitride (ISTSN)

On-Going Research (Graduate Student - G. Raghavendra)

Purpose

To study the high temperature high strain rate properties of *in situ* reinforced silicon nitride (IRSN), involving both single loading failure modes and high strain rate fatigue.

Experimental Goals

1. Measure stress strain data for IRSN at temperatures ranging from 25 to 1200°C.
2. Develop high temperature fatigue testing procedures.
3. Use ultrasonic techniques to measure elastic modulus of the material and assess degradation of elastic modulus with loading cycles.

Experimental Techniques Developed

1. *Simplified Specimen Geometry* reduces chipping of specimen ends and premature failure in the gage section. Experiments showed that a cylindrical specimen was least prone to chipping.

2. *Thorough Specimen Preparation* further reduces the tendency for premature failure. All specimens were polished to a 1 micron finish using a STRUERS automatic polisher, and, only specimens with flat and parallel faces were used for testing.
3. *Confined Tungsten Carbide (WC)* Platen system is used to prevent premature failure of the specimen-platen interface. The WC inserts are impedance matched with the bars and confined in a thin walled Inconel 625 cylinder. The confinement increases the failure strength of WC substantially and prevents failure of the platens during the experiment.
4. *Bar Movers* are particularly important at higher temperatures of testing as they ensure that the bars are kept at relatively low temperatures during the high temperature experiment.
5. *Multi-stepped Strikers* allow multiple fatigue cycles in one event. Special double tapered striker bars have been developed.
6. *Ultrasonic Measurement Techniques* have been developed to measure the elastic modulus of the material. In future tests, the elastic modulus will be measured periodically after some number of loading cycles.

Differential Strain Measurement

The strain measurement in a high strain rate tests is obtained using strain gages mounted on the incident bar. This measurement not accurate because of an interface error which arises due to the high stiffness of the ceramic. Strain gages cannot be mounted directly on the specimen for two reasons :

(a) small size of the specimens makes it difficult to mount strain gages onto the specimen surface.

(b) the adhesives used to mount the gages cannot be used at elevated temperatures.

A technique has been developed wherein two specimens of different lengths are tested and the interface error is determined and corrected. After careful calibration, it is possible to correct the strain data directly obtained from the strain gage of the incident bar without any additional measurements or tests.

Results of the Experimental Work

- *Ultrasonic Measurements*

The longitudinal wave speed through *in situ* reinforced silicon nitride was found to be 10,896 m/s. The density of the material being 3,000 kg/m^3, the elastic modulus of the material was found to be 342 GPa.

- *Dynamic tests*

Room-temperature tests:

Specimens were tested elastically to stresses of around 2.76 GPa (400ksi) at room temperature. The tests were conducted at two strain rates -700/s and 1,700/s.

* The DSM technique was applied on elastic tests at room temperature and the elastic modulus was determined. The value obtained was 350GPa (for a strain rate of 700/s), which correlates well with the ultrasonic value.

* The failure stress at room temperature at a strain rate of 1,700/s was found to be 5.17 GPa (750 ksi).

Tests over a range of temperatures:

High strain-rate tests were conducted over a range of temperatures from room temperature to 830°C. Both elastic tests as well as tests to failure were carried out. At each temperature, the elastic modulus of the material was determined by using the DSM technique.

* The elastic modulus was found to decrease with increase in temperature (see Figure 1, page 6).
* The failure stress was found to decrease with increase in temperature (see Figure 2, page 6).
* A change in modulus was observed just prior to failure at elevated temperatures (above 630°C).
* Some inelastic deformation was observed at elevated temperatures (above 630°C) at stress levels of 2.5 GPa and higher.
* All specimens shattered during failure.
* Specimens that were not polished properly failed prematurely.
* Premature failure of the platens led to premature failure of the specimen in one test.
* At 830°C, oxidation of the platens occurred. A green-blue deposit was found on the platen surface after the test. The region where the specimen was in contact with the platen was not oxidized.

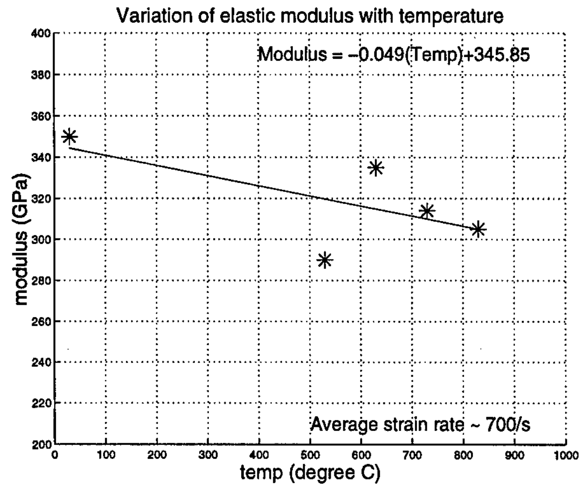

Figure 1: Plot showing variation of elastic modulus with temperature

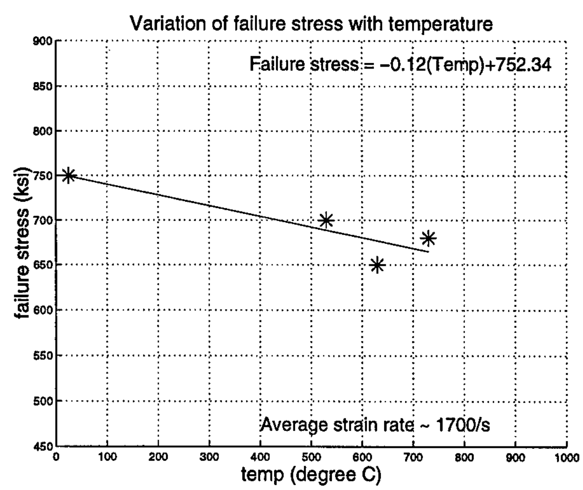

Figure 2: Plot showing the variation of failure stress with temperature

2.5 Microstructural Study of Fractured Silicon Nitride Samples

On-going research (Graduate Student - G. Raghavendra)

Purpose

To microstructurally characterize the material both before testing and after testing, in order to understand the influence of different microstructural features on the failure mechanisms and the toughening mechanisms in *in situ* reinforced silicon nitride.

Primary Characterization

The material was characterized prior to any testing, using SEM, XRD and high resolution TEM. The work showed that the material consisted of large acicular β Si_3N_4 grains and small equiaxed α Si_3N_4 grains, with an intergranular boundary phase. Details of the microstructural features may be obtained from Liu & Nemat-Nasser, *Materials Science & Engineering A*, (1998) 242-252.

Characterization after Testing

The specimens that failed during high temperature testing, at high strain rates (1700/s), shattered during the test. The powders were examined in the SEM and the TEM. The following observations were made:

The failure was predominantly inter-granular. This was concluded from both SEM studies, where the crack-path was found to be clearly intergranular, and, from TEM studies, where the individual particles were found to be grains.

There was evidence of grain pull-out. In the SEM figures 1 & 2 (page 8), there are clearly regions that were occupied by β grains, that, have been `pulled-out', as a result of the loading. This suggests thatbgrain pull-out is a toughening mechanism in *in situ* reinforced silicon nitride.

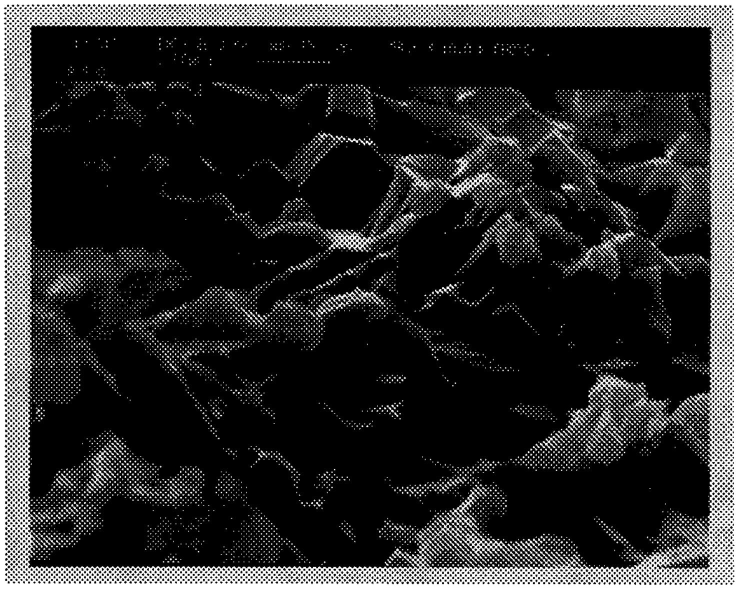

Figure 1: SEM image showing grain-pull out; the acicular β grains have been pulled out.

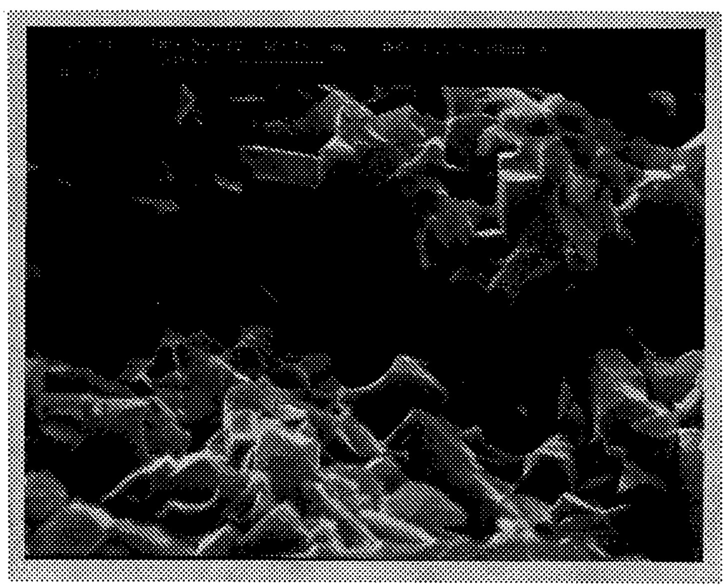

Figure 2: SEM image showing the crack path in *in situ* reinforced silicon nitride.

3. LIST OF ALL PUBLICATIONS

Published

- "The Microstructure and Boundary Phases of *In-Situ* Reinforced Silicon Nitride," M. Liu and S. Nemat-Nasser, *Materials Science and Engineering A.*, Vol. 254, No. 1-2, (1998) 242-252. (also supported by ARO DAAH04-95-7-0369 to UCSD.

Accepted (In-Press) or Submitted

- "Microstructure of a Bearing-Grade Silicon Nitride," M. Liu and S. Nemat-Nasser, *Journal of Materials Research,* submitted 3/99.
- "Dynamic Properties of *in situ* Reinforced Silicon Nitride at Elevated Temperatures," G. Raghavendra, S. Nemat-Nasser, and M. Liu, *Materials Science and Engineering A*, 1999 TMS Annual Conference, Symposium to honor Ali S.Argon, San Diego, CA. March 23, 1999, in-press.

4. PARTICIPATING SCIENTIFIC PERSONNEL

Mingqui Liu (9/95 - 11/97) (partially supported) Postgraduate Researcher. Research focused on grain-boundary structures of bearing grade silicon nitride and *in situ* reinforced silicon nitride.

Gayathri Raghavendra (10/96 - present) Graduate Student Researcher. Research focuses on the measuring of the stress strain data for *in-situ* reinforced silicon nitride at temperatures ranging from 25 to 1200°C; developing high temperature fatigue testing procedures, and using ultrasonic techniques to measure elastic modulus of the material and assess degradation of elastic modulus with loading cycles.

Degree Conferred: MS by examination, December 1997

Vinod Sharma (6/96 - 3/97) Postgraduate Researcher. Research focused on the response and failure modes of silicon nitride under compression fatigue.

5. REPORT OF INVENTIONS (BY TITLE ONLY): NONE

Dynamic Properties of *in situ* Reinforced Silicon Nitride at Elevated Temperatures

Gayathri Raghavendra, Sia Nemat-Nasser*and Mingqi Liu

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Abstract

This study is aimed at evaluating the compressive dynamic response of *in situ* reinforced silicon nitride at elevated temperatures. The material has been characterized before experimentation microstructurally [1]. A unique experimental facility, the modified split Hopkinson pressure bar, has been developed for the purpose of conducting high strain-rate tests at elevated temperatures. A new measurement technique, called Differential Strain Measurement (DSM) has been developed to obtain accurate values of strain. Extensive tests have been performed over a range of temperatures, and these tests show that the elastic modulus and the failure stress of the material decrease with increasing temperature.

Keywords: Silicon nitride, high strain-rate, elevated, temperature.

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1 Introduction

Silicon nitride has a unique combination of mechanical and thermal properties that make it an ideal material for high-temperature structural applications; [2], [3], [4]. Some of the important applications of silicon nitride are heat engine components and cutting tool inserts. Many moving components are subjected to dynamic loading and, hence, it is necessary to study the dynamic response of the material over a broad range of temperatures. It is also important to relate the dynamic response and failure modes to the microstructure, as the material performance can be significantly improved by appropriate microstructural design. The purpose of this study is to evaluate the compressive dynamic response of the material, over a range of temperatures.

2 Experimental Procedure

2.1 Material

The material used for this study is *in situ* reinforced silicon nitride (AS800), provided by Allied Signal Inc. It is produced from silicon nitride powders sintered at a temperature above 1,750^oC

in a nitrogen atmosphere with a gas pressure of about 20.7 MPa. The sintering aids are La_2O_3, Y_2O_3, and a small amount of SrO. After sintering, a post sintering treatment is performed.

2.2 Microstructure

A preliminary microstructural study has been carried out on this *in situ* reinforced silicon nitride using High Resolution Transmission Electron Microscopy (HRTEM), X-Ray Diffraction (XRD), Selected Area Electron Diffraction (SAED) and Scanning Electron Microscopy (SEM); see [1].

2.3 Ultrasonic measurement

To measure longitudinal wave speed in the material, ultrasonic tests are conducted on a Matec MB 800 system along with a Marconi Instruments signal generator and a Tektronix dual-beam oscilloscope. The Young's modulus (E) of the material is related to the longitudinal wave speed V_L by:

$$E = {V_L}^2 \rho \frac{(1+\nu)(1-2\nu)}{(1-\nu}, \qquad (1)$$

where, ρ is the density ($3300 kg/m^3$) and ν is the Poisson ratio (0.222).

2.4 Dynamic testing

Dynamic tests are carried out over a range of temperatures, using a modified split Hopkinson compression bar. In the classical split Hopkinson compression bar ([5], [6]), the tensile pulse which reflects off the incident bar and the sample interface, is in turn reflected from the free end of the incident bar as a compressive pulse and reloads the sample. Hence, this technique suffers from the drawback of uncontrolled reloading of the sample. To overcome this problem, a modified split Hopkinson compression bar apparatus is used ([7]).

Another modification to this instrument is the inclusion of impedance- matched tungsten carbide (WC) inserts between the sample and the bars. The main purpose of the inserts is to prevent the yielding of the maraging steel bars during a test, when the sample is subjected to very high stress levels [8]. At elevated temperatures of testing, it is found that the inserts themselves fail at the high stress levels. To overcome this, the inserts are confined by a thin- walled cylinder of Inconel. Figure 1 shows the details of the modified split Hopkinson compression bar apparatus, and Figure 2 shows the details of the WC inserts.

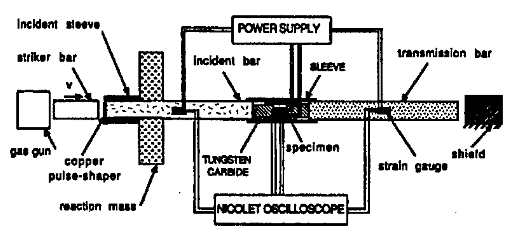

Figure 1: The modified split-Hopkinson pressure bar for dynamic tests at elevated temperatures

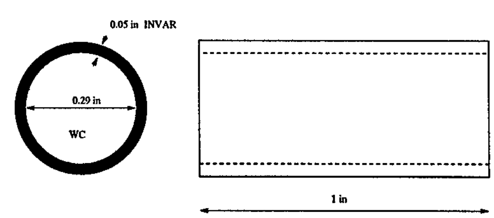

Figure 2: Schematic of the WC inserts used for recovery dynamic testing at elevated temperatures

To produce a constant strain rate in the sam-

ple, a copper pulse shaper is used at the striker-incident bar interface, to generate ramped pulses ([8]). Details for choosing an appropriate geometry and dimension of the pulse shaper are given in [7].

2.5 Strain-measurement technique

An important issue in the dynamic testing of hard ceramics, such as silicon nitride, is the ability to accurately measure strains in the samples. The problem arises due to the high effective impedance of the material, which results in a small reflected pulse [8]. In addition, because of their high hardness, the samples indent the loading platen and the measured strain is usually many times greater than the actual sample strain. At room temperature, this problem can be overcome by mounting strain gages on the sample and measuring the strain directly as the sample is deforming. Strain gages, however, have a limited temperature range of operation and cannot be used at elevated temperatures (above 500^oC). A new technique which we call Differential Strain Measurement (DSM) has been developed to obtain accurate and reliable measurements of strains for dynamic tests at elevated temperatures.

The (DSM) technique is based on the fact that an interface error is introduced due to the indentation of the bars by the sample. It turns out that the interface error is essentially constant for a given cross-sectional area of contact between the sample and the bars. Two high strain-rate tests are conducted on the Hopkinson bar with two samples of different lengths, but of the same cross-sectional area. By subtracting the displacements obtained in the two cases, it is possible to determine the interface error. After careful calibration, it becomes possible to determine the actual stress-strain data from the stress-strain data obtained from a test, by accounting for the interface error.

3 Results and Discussion

3.1 Microstructure

The material consists of approximately 90% silicon nitride and 10% second phase at the grain boundaries; [1]. Figure 3 is an SEM micrograph of the polished surface of the AS800 silicon nitride specimen, in the unetched condition. The grain structure can be clearly seen after the specimen is etched at 400^OC in molten NaOH for 4-6 min. As shown in Figure 4, AS800 silicon nitride consists primarily of acicular grains. Due to the high sintering temperature, grains with different widths and lengths are formed. The average grain width is approximately $0.8\mu m$ and the aspect ratio is greater than 4. This morphology is typical for α/β mixtures.

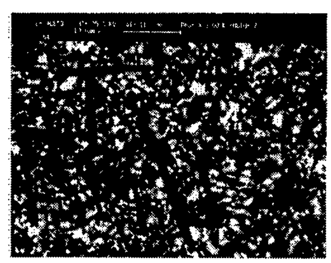

Figure 3: SEM micrograph of the polished surface of AS800 silicon nitride - Boundary phase (white) is extensively seen at the grain pockets

Figure 5 is an SEM fractograph of AS800 silicon nitride subjected to high strain-rate shear, revealing a typical interlocking microstructure, which is highly resistant to deformation. Under compressive loads, the acicular grains inter-

Figure 4: SEM micrograph of the polished and etched surface of AS800 silicon nitride - the boundary phase has been etched away, leaving acicular silicon nitride grains

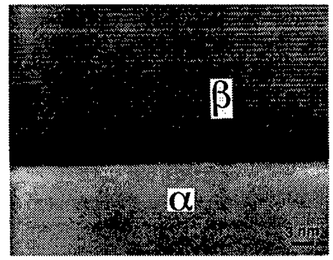

Figure 6: HRTEM micrograph of AS800 silicon nitride showing an amorphous grain boundary phase

lock, resulting in increased creep resistance by inhibiting grain boundary sliding. Under tensile stresses, in addition to limiting grain boundary sliding, the elongated grains improve the stress rupture properties by bridging the microcracks; [9].

Figure 5: SEM micrograph of AS800 silicon nitride with an interlocking microstructure

Figure 6 is a high-resolution electron micrograph taken at a planar grain boundary region, showing an amorphous phase at a two-grain boundary. The thickness of the boundary phase is approximately 1.8nm.

3.2 Ultrasonic measurements

The longitudinal wave speed through *in situ* reinforced silicon nitride is found to be 10,896 m/s. The density of the material being $3,000 kg/m^3$, the elastic modulus of the material is found to be 342 GPa.

3.3 Dynamic testing

3.3.1 Room-temperature tests

Specimens are tested elastically to stresses of around 2.76 GPa (400 ksi) at room temperature. Tests are performed using the set up described in the earlier sections. The tests are conducted at two strain rates - 700/s and 1,700/s.

- The DSM technique is applied on elastic tests at room temperature and the elastic modulus is determined. The value obtained is 350GPa (for a strain rate of 700/s), which correlates well with the ultrasonic value.
- The failure stress at room temperature at a strain rate of 1,700/s is found to be 5.17 GPa (750 ksi).

3.3.2 Tests over a range of temperatures

High strain-rate tests are conducted over a range of temperatures from room temperature to 830^oC. Both elastic tests as well as tests to failure are carried out. At each temperature, the

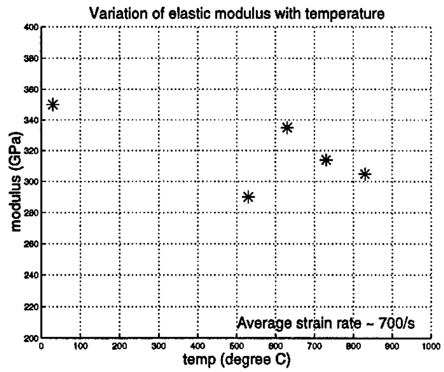

Figure 7: Plot showing variation of elastic modulus with temperature

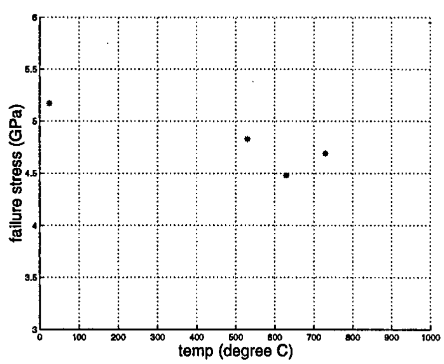

Figure 8: Plot showing the variation of failure stress with temperature

elastic modulus of the material is determined by using the DSM technique.

- The elastic modulus is found to decrease with increase in temperature - Figure 7.
- The failure stress is found to decrease with increase in temperature - Figure 8.
- A change in modulus is observed just prior to failure at elevated temperatures (above 630^oC) - Figure 9.
- Some inelastic deformation is observed at elevated temperatures (above 630^oC) at stress levels of 2.5 GPa and higher - Figure 10.
- All specimens shatter during failure.
- Specimens that are not polished properly fail prematurely.
- Premature failure of the platens led to premature failure of the specimen in one test.
- At 830^oC, oxidation of the platens occurs. A green-blue deposit is found on the platen surface after the test. The region where the specimen is in contact with the platen is not oxidized.

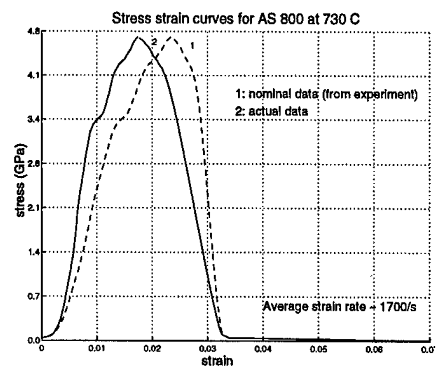

Figure 9: Stress-strain data for *in situ* reinforced silicon nitride, tested to failure at a strain rate of 1,700/s at 730^oC - note that there is a decrease in modulus prior to failure (test data has been corrected using the DSM technique)

The two important mechanisms which are applicable to crack growth in silicon nitride in the present testing scenario, are the Surface Roughness Mechanism ([10], [11], [12], [13] and [14]) and Grain Boundary Sliding ([15], [16], [17] and [18]). Over the range of temperatures and strain rates of testing in this study, it is possible that more than one mechanism is operative. Depending on the regime, one mechanism may be more dominant than the other. Of particular interest is the fact that a change in elastic modulus is observed

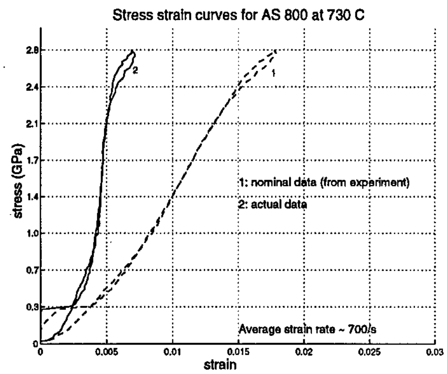

Figure 10: Stress-strain data for *in situ* reinforced silicon nitride, tested to 2.5 GPa at a strain rate of 700/s at 630^oC - note that there is some inelastic deformation at stress levels of 2.5 GPa and higher (test data has been corrected using the DSM technique)

just prior to failure at the elevated temperatures. This non-linear deformation behavior may be attributed to the ongoing accumulation of creep damage (micro-cavities and micro-cracking) during dynamic testing. Similar observations have been made in [19] and [20], where the response of silicon nitride over a range of stress rates and temperatures is studied. It is suggested that the minor deviation from linearity just prior to failure is a result of compliance changes due to subcritical crack growth of the failure -inducing flaw. Damage accumulation by formation and growth of creep cavities and micro-cracks could cause a decrease in Young's modulus and lead to an apparent non-linear stress-strain behavior [21, 22]. The non-linear deformation could be attributed to creep processes related to diffusion, viscous flow or other mechanisms or a combination of these.

Acknowledgement: This work is supported by a grant from the Army Research Office (ARO) - Grant number DAAH04-95-1-0609 to the University of California, San Diego.

References

[1] Mingqi Liu and S. Nemat-Nasser. The microstructure and boundary phases of in-situ reinforced silicon nitride. *Materials Science and Engineering A*, A254(1-2):242–52, 1998.

[2] R. A. L. Drew. Silicon nitride and sialon ceramics - a review. *Canadian Metallurgical Quarterly*, 27(1):59–64, 1988.

[3] K. H. Jack. Sialon ceramics - retrospect and prospect. *Materials Research Society*, pages 15–27, 1993.

[4] V. K. Pujari and M. Abouaf. Cost effective reliable silicon nitride for high performance structural applications. *Key Engineering Materials*, 132-1361:342–345, 1997.

[5] P. S. Follansbee. The Hopkinson bar. *Metals Handbook*, 8:198–203, 1985.

[6] U. S. Lindholm. Some experiments with the split Hopkinson pressure bar. *Journal of the Mechanics and Physics of Solids*, 12:317–335, 1964.

[7] S. Nemat-Nasser, J. B. Isaacs, and J. E. Starrett. Hopkinson techniques for dynamic experiments. *Proceedings of the Royal Society of London*, 435 A:371–391, 1991.

[8] V. Sharma, S. Nemat-Nasser, and K. S. Vecchio. Dynamic compression fatigue of hot-pressed silicon nitride. *Experimental Mechanics*, 21:351–323, 1994.

[9] C. W. Li, S. C. Lui, and J. Goldacker. Relation between strength, microstructure, and

grain-bridging characteristics in in-situ reinforced silicon nitride. *Journal of the American Ceramic Society*, 78(2):449–59, 1995.

[10] S. Nemat-Nasser and H. Horii. Compression-induced non-planar crack extension with application to splitting, exfoliation and rockburst. *Journal of Geophysical Research*, 87(88):6805–21, 1982.

[11] H. Horri and S. Nemat-Nasser. Compression induced microcrack growth in brittle solids : Axial splitting and shear failure. *J. Geophys. Res.*, 90:3105–3125, 1985.

[12] H. Horii and S. Nemat-Nasser. Brittle failure in compression : Splitting, faulting and brittle-ductile transition. *Philosophical Transactions of the Royal Society of London*, 319:337–374, 1986.

[13] L. Ewart and S. Suresh. Crack propagation in ceramics under cyclic loads. *Journal of Materials Science*, 22:1173–1192, 1987.

[14] Subra Suresh and L. X. Han. Fracture of silicon nitride- SiC reinforced composites under cyclic loads. *Journal of the American Ceramic Society*, 71(3):C–158–C–161, 1988.

[15] A. G. Evans and S. M. Wiederhorn. Crack propagation and failure prediction in silicon nitride at elevated temperatures. *Journal of Materials Science*, 9(2):270–78, 1974.

[16] U. Ramamurty, T. Hansson, and S. Suresh. High-temperature crack growth in monolithic and SiC-w reinforced silicon nitride under static and cyclic loads. *Journal of the American Ceramic Society*, 77(11):2985–99, 1994.

[17] T. Hansson, U. Ramamurty, C. Bull, and R. Warren. Elevated temperature fracture behavior of monolithic and SiC-w - reinforced silicon nitride under quasi-static loads. *Materials Science and Engineering*, A209:137–148, 1996.

[18] G. Bernard-Granger, J. Crampon, R. Duclos, and B. Cales. High temperature creep behavior of beta-silicon nitride alpha sialon ceramics. *Journal of the European Ceramic Society*, 17(13):1647–54, 1997.

[19] C. J. Lin, M. G. Jenkins, and M. K. Ferber. Tensile dynamic and static fatigue relations for a HIPed silicon nitride at elevated temperatures. *Journal of the European Ceramic Society*, pages 3–13, 1993.

[20] D. E. McCullum, N. L. Hecht, L. Chuck, and S. M. Goodrich. Summary of results of the effects of environment on mechanical behavior of high-performance ceramics. *Ceramic Engineering Science Proceedings*, 12:1886–1913, 1991.

[21] A. Venkateswaran, K. Y. Donaldson, and D. P. H. Hasselman. Role of intergranular damage-induced decrease in Young's modulus in the non-linear deformation and fracture of alumina at elevated temperatures. *Journal of the American Ceramic Society*, 71:565–576, 1988.

[22] D. P. H. Hasselman, A. Venkateswaran, and Donaldson. K. Y. Contribution of damage by multiple crack growth to the strain rate sensitivity of a polycrystalline alumina at elevated temperatures. *Journal of Materials Science*, 24:671–680, 1989.